Definition

"Drying is a mass transfer process in which water or another solvent is removed by evaporation from a solid, semi-solid or liquid."

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Objectives

1. Lack of understanding about the impact of the presence of moisture, environmental conditions and drying process parameters on drugs, critical product quality attributes, etc., creates challenges during product development, manufacturing, storage and use.
2. In order to improve and retain product quality such moisture sensitive products need to processed to remove solvents using drying.
3. The main objective of drying is to preserve pharmaceutical products and increase their shelf life by reducing the water content and water activity.

Its other objectives are to:

1. carryout size reduction;
2. avoid deterioration and the need for use of refrigeration systems for transport and storage, which is expensive;
3. to reduce space requirements for storage and transport;
4. to minimize chemical changes, such as enzymatic and non-enzymatic browning, and to maximize therapeutic activity retention of bioactive compounds during drying.

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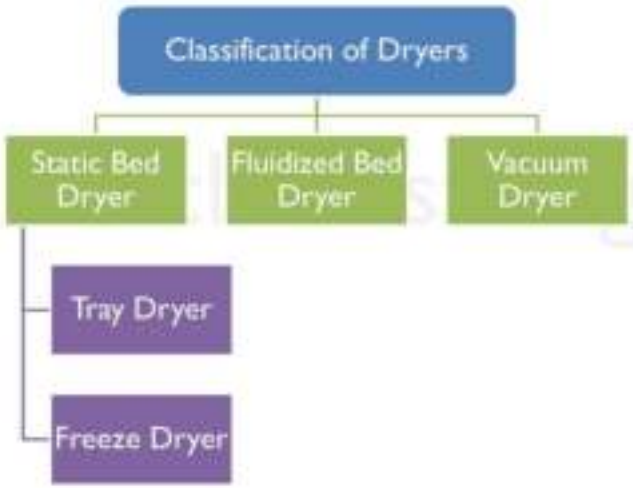
Classification of Dryers



Fluidized Bed Dryer

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Fluidized Bed Dryer

1. Fluidized bed dryer (FBD) is well known and widely used equipment in the pharmaceutical manufacturing.
2. It is used in granulation process to attain desired moisture levels in the granules or powders required for perfect compression of tablet formulations.
3. Conventional FBDs include batch fluidized bed dryer, semi-continuous fluidized bed dryer, well-mixed continuous fluidized dryer and plug flow fluidized bed dryer.
4. Fluidized bed dryer has a high drying rate and the material is dried in a very short time.
5. Material remains free- flowing and uniform.

Principle:

1. The FBD works on a principle of fluidization of the feed materials.
2. In this process, hot air is introduced at high pressure through a perforated bed of moist solid particulate.
3. If air is allowed to flow through a bed of solid material in the upward direction with the velocity greater than the settling rate of the particles, the solid particles are blown-up and remain suspended in the air stream.
4. Heat transfer is accomplished by direct contact between the wet solid and hot gases.
5. Use of hot air to fluidizing the bed increases the drying rate.

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Construction:

1. A FBD contains a stainless steel chamber having a removable perforated bottom known as the bowl.
2. A typical FBD, Fig. 4.17, consists of the air handling unit, product container, exhaust filter, exhaust blower, control panel, air distribution plate, spray nozzle, and solution deliver.
3. The appropriate choice of distributor used during drying process ensures uniform and stable fluidization.
4. The pressure drops across the distributor must be high enough to ensure good and uniform fluidization.

Working:

1. Material to be dried is placed in the bowl type vessel.
2. Air is introduced from the top and heated at required temperature by the heaters.
3. The air is filtered through the filter and then passes through the bed of the material at the bottom.
4. The airflow is generated by the fans fitted at the top of the equipment.
5. The air flow rate and the operating temperature are adjusted by the control panel.
6. As the flow of air increases, the bed expands and particles of powder start to rise up in a turbulent motion.
7. The regular contact with air causes the material to dry.

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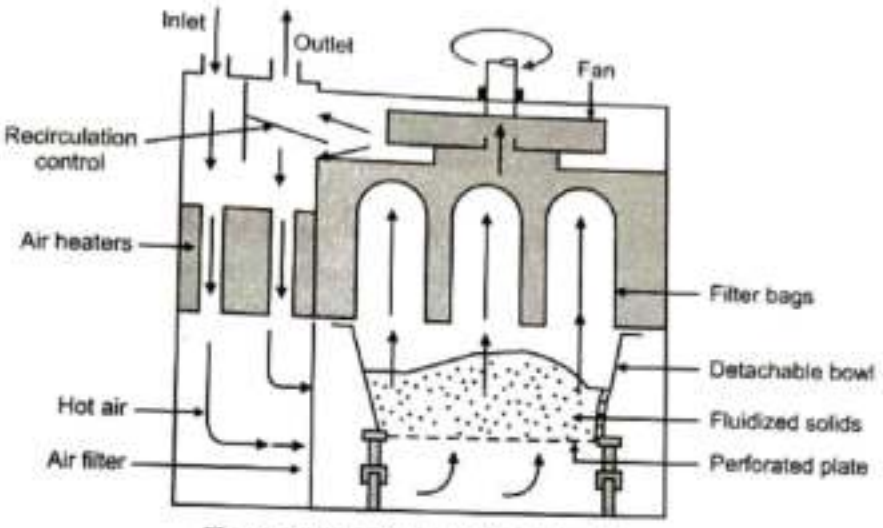

Fig. 4.17: Vertical Fluidized Bed Dryer

8. The air leaving the FBD passes through the filter to collect the fine particles of the material.
9. FBD bags have finger-like shape to increase the volume of the drying bed that helps to increase the drying rate and decrease the drying time.
10. The vaporized liquid is carried away by the drying gases. Sometimes to save energy, the exit gas is partially recycled.

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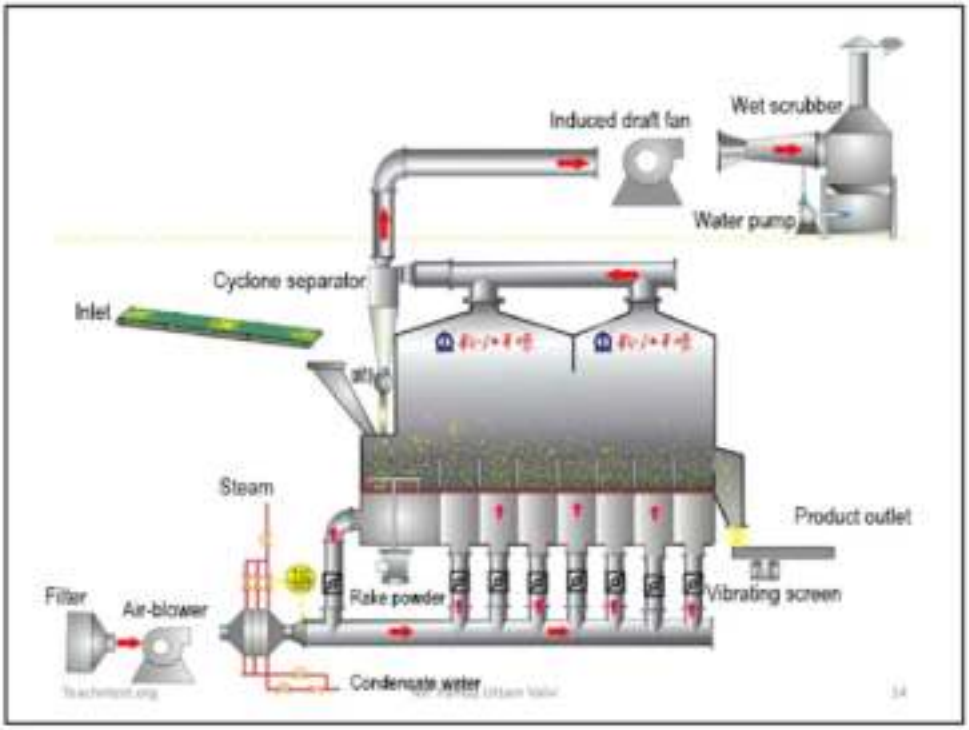

Advantages:

1. It takes less time to complete drying as compared to other dryer.
2. Drying is achieved at constant rate.
3. Handling time is also short
4. It is available in different sizes with the different drying capacity.
5. The equipment is simple and less labour costs required.
6. More thermal efficiency.
7. Drying capacity is more than other dryer.
8. It facilitates the drying of thermolabile substances since the contact time for drying is short.
9. It is batch type or continuous type process.

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Disadvantages:

1. Many organic powders develop electrostatic charges during drying. To avoid this efficient electrical grounding of the dryer is essential.
2. Chance of attrition of some materials resulting in the production of fines.

Applications:

1. FBD is used for drying and mixing of powders and agglomeration of materials.
2. It is used in granulation and coating of powders, granules, tablets, pellets, beads.
3. It is used as fluidized bed reactors, for solids separation and for heat/mass transfer.
4. The modified FBDs are used in precision granulation, top spray granulation, spray drying granulation and granulation coating.

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Freeze Drying

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Freeze Drying

1. Freeze drying, also called lyophilization, is a process in which water is frozen, followed by its removal from the sample, initially by sublimation followed by desorption.
2. The equipment used to dry solutions or suspensions at or below freezing points of liquids is called as freeze dryer or lyophilizer.
3. Freeze drying is used in the manufacture of pharmaceuticals and biologicals that are thermolabile or otherwise unstable in water or moisture for prolonged storage periods, but that are stable in the dry state.

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Principle:

1. The principle involved in freeze drying is sublimation, where water passes directly from solid state (ice) to the vapour state without passing through the liquid state.
2. Sublimation of water can take place at pressures and temperature below triple point of water.
   - (The temperature and pressure at which a substance can exist in equilibrium in the liquid, solid, and gaseous states. The triple point of pure water is at 0.01 °C and 4.58 mm Hg.)
3. The material to be dried is first frozen and then subjected under a high vacuum to heat (by conduction or radiation or by both) so that frozen liquid sublimes leaving only non-volatile solid, dried components of the original liquid.

Construction:

1. Generally, there are three types of freeze dryers, for example, manifold freeze-dryer, the rotary freeze dryer and the tray style freeze-dryer.
2. These freeze-dryers differ in the method by which the dried substance is interfaced with a condenser.
3. The components common to all of them are a vacuum pump to reduce the ambient gas pressure and a condenser to remove the moisture by condensation on a surface cooled to -20 to -80 °C.
4. A freeze dryer consists of a vacuum chamber wherein products to be dried are kept on shelves and capable of cooling and heating containers and their contents.
5. A vacuum pump, a refrigeration unit, and associated controls are connected to the vacuum chamber.

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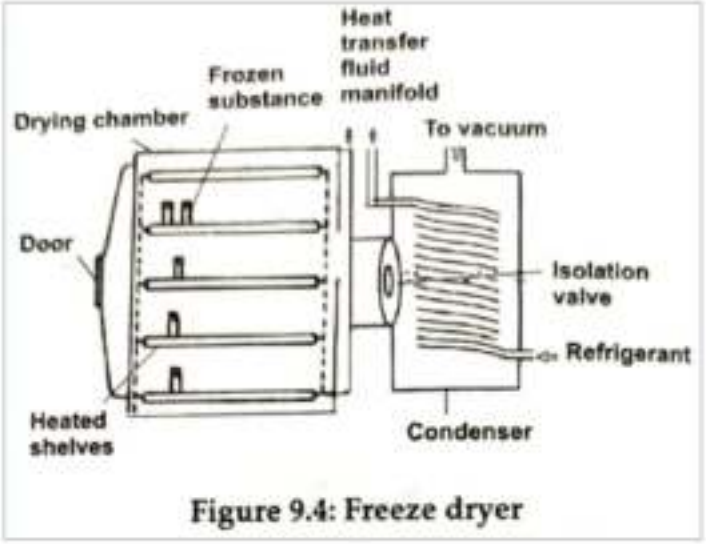

Figure 9.4: Freeze dryer

Working:

1. Traditional freeze drying is a complex process that requires a careful balancing of sample, equipment and processing techniques.
2. In this process, water is removed from a sample after it is frozen and placed under a vacuum, allowing the ice to change directly from solid to vapour without passing through a liquid phase.
3. It is performed at temperature and pressure conditions below the triple point of liquid, to enable sublimation of frozen material.
4. The entire process is performed at low temperature and pressure. Steps involved in lyophilization start from sample preparation followed by freezing, primary drying and secondary drying, to obtain the final dried product.

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5. The vapour pressure of water increases with an increase in temperature during the primary drying.
6. Therefore, primary drying temperature should be kept as high as possible, but below the critical process temperature, to avoid a loss of cake structure.
7. There are four important stages in the complete freeze drying process namely pretreatment, freezing, primary drying and secondary drying.

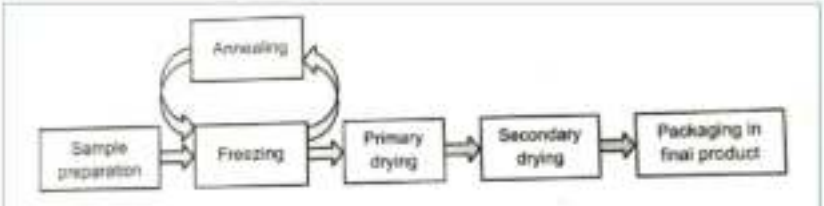
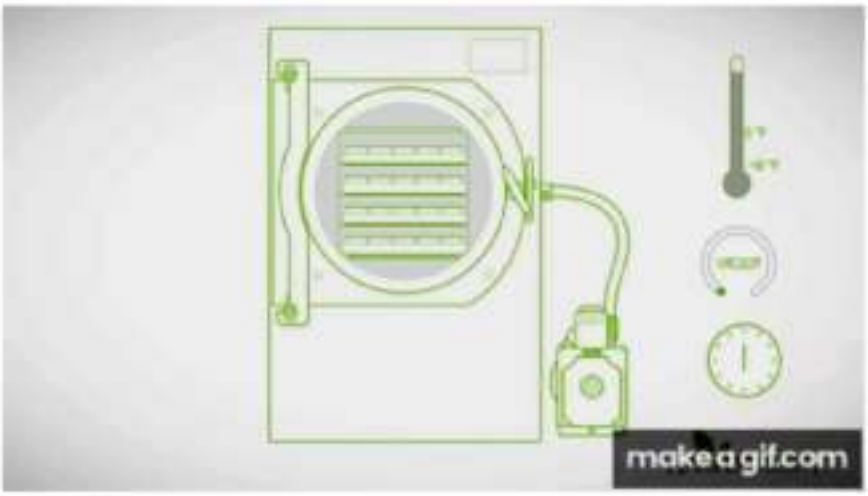


Fig. 4.18: Freeze Drying Cycle- Sequence of Operational Steps



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1. **Pretreatment:**

   In this stage product is treated for freeze concentration, solution phase concentration, preserve product appearance, stabilize reactive products, prior to freezing increase surface area, and decrease high vapour pressure solvents concentration.

2. **Freezing:**

   During freezing stage usually the liquid sample is cooled down to -40 to -60 °C until pure crystalline ice forms from part of the liquid and the remainder of the sample is freeze-concentrated into a glassy state where the viscosity is too high to allow further crystallization.

3. **Primary drying:**

In primary drying the ice formed during the freezing is removed by sublimation under vacuum at low temperatures, leaving a highly porous structure in the remaining amorphous solute, that is typically 10% water. This step is carried out at pressures of 104 to 109 atmospheres, and a product temperature of -45 to -20 °C. The sublimation during primary drying is the result of coupled heat- and mass-transfer processes.

4. **Secondary drying:**

This is last step wherein most of the remaining water is desorbed from the glass as the temperature of the sample is gradually increased up to 10 - 15 °C while maintaining low pressures. Ideally, the final product is a dry cake with a high surface area and low moisture content (<3% w/w) which can be easily reconstituted.

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Steps in Freeze Drying Process

Advantages:

1. This is suitable for drying heat sensitive products.
2. Freeze dried product is porous and easy to be rehydrated and instantly dissolved.
3. Drying takes place at very low temperature, so that enzyme action is inhibited and chemical decomposition, particularly hydrolysis is minimized.
4. Denaturation of protein does not occur.
5. Loss of volatile materials is less.
6. Sterility can be minimized.

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Disadvantages:

1. The process is very slow.
2. Expensive process.
3. The period of drying is high.
4. The product is prone to oxidation, it must be vacuum packed.

Applications:

1. Freeze-drying is used to increase the shelf life of thermolabile products, such as vaccines and other injectable.
2. It is used to enhance stability of products during storage, shipping, and transportation.
3. Freeze-drying is used to reduce weight of products.
4. It is used to preserve blood products in freeze-dried form.
5. It is used in chemical synthesis to make products more stable and easier to dissolve in water.
6. Freeze-drying can effectively be used in bio-separations in purification procedures.
7. It can be used to concentrate low molecular weight substances that are too small to be removed by a membrane filtration.

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Reference

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